



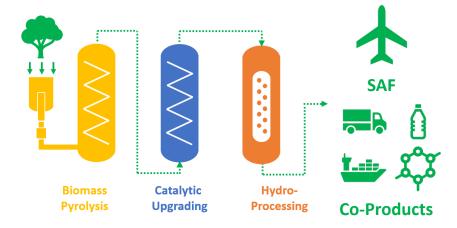
Catalytic Upgrading of Pyrolysis Products for the Production of Sustainable Aviation Fuel

Michael Griffin Catalytic Upgrading Session BETO Peer Review April 3-7, 2023



Project Overview

Project Goal: Develop a technology pathway to convert woody biomass into sustainable aviation fuel (SAF) and other biogenic products via catalytic fast pyrolysis (CFP) and hydrotreating



Key Advantage: Catalytic fast pyrolysis generates a stabilized bio-oil that de-risks down-stream hydroprocessing

Market Trends: Repurposing refinery hydroprocessing infrastructure for the production of renewable diesel and sustainable aviation fuel from fats, oils, and greases

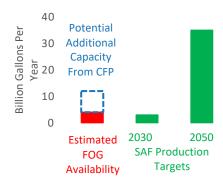
Phillips 66: Rodeo, CA



Shell: Rotterdam, Netherlands



Existing approach is constrained by the availability of waste fats, oils, and greases



This research opens pathways for SAF production from forest resources and woody wastes

Estimated Availability of Forest Resources and Woody Wastes

133 Million Dry Tons/Yr

8 BGPY
Hydrocarbon Fuel Potential

DOE Billion Ton Report, 2016, ORNL/TM-2016/160



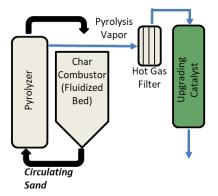
Project Overview

Previous Research: Fixed Bed Catalytic Fast Pyrolysis

- Demonstrated potential to achieve 78% GHG emissions reduction with a modelled minimum fuel selling price (MFSP) < \$3.00 GGE
- Evaluated durability over 100+ reaction regeneration cycles
- Identified opportunities to valorize high value co-product streams

Challenges:

- Requires co-fed hydrogen and utilizes noble metal catalysts
- Rapid deactivation necessitates multiple reactors for continuous upgrading
- Poor heat transfer in fixed bed



Dutta. A., et al., J. Chem. Eng., 2023, 451, 138485 French, R. J., et al, ACS Sustain. Chem. Eng., 2021, 9, 1235 Griffin, M.B., et al. Ener. Environ. Sci., 2018, 11, 2904

2021 Pivot

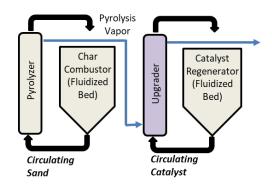
Current Research: Fluidized Bed Catalytic Fast Pyrolysis

Advantages:

- Can be performed without co-fed hydrogen using zeolite catalysts
- Rapid deactivation can be managed through continuous regeneration
- Improved heat transfer in fluidized bed

Research Needs:

- Benchmark performance for SAF production
- Establish critical material attributes for CFP-oil
- Increase carbon efficiency and fuel yield





Approach: Schedule

Kickoff 10/1/21

Phase 1: Current Work

Go/No Go 06/30/23

Phase 2: Future Work

End Date 9/30/24

Objectives:

Benchmark performance for SAF production and establish critical material attributes for CPF-oil

Technical Targets:

- Demonstrate the production of SAF that meets
 ASTM D4054 guidelines for density, viscosity,
 boiling point, heating value, flash point, freeze
 point, and volatility.
- Demonstrate ≥70% GHG reduction without requiring noble metal catalyst or co-fed hydrogen during CFP
- Establish three critical material attributes (CMAs) for CFP bio-oil

Objectives:

Increase process carbon efficiency and fuel yields

- Modification of technical CFP catalysts to reduce coking and increase bio-oil production
- Process optimization to effectively utilize byproduct streams (e.g., gasses, volatile condensables, char)

Technical Targets

- Demonstrate a ≥ 20% increase in carbon efficiency to SAF while maintaining the ability to meet the fuel property, GHG reduction, and CMA targets outlined in the project's GNG.
- Demonstrate hydroprocessing durability for ≥ 500 h
 time on stream



Approach: Integrated Experimental Campaigns

Biomass Handling and Preprocessing (INL)



Ex-Situ CFP

Fluidized Bed ZSM-5 SAR 30



Hydrotreating

Fixed Bed Sulfided NiMo/Al₂O₃



Fractionation and Fuel Property Analysis

- Continuous CFP experiments performed without requiring cofed hydrogen or noble metal catalysts
- Realistic biomass feedstocks including clean pine and waste forest residues
- Sufficient product volume for fuel property assessment
- Technical catalyst formulations for CFP and hydrotreating steps
- Comprehensive analytical capabilities to achieve material balances of 100 ± 5%

Supporting Analysis

Pre- and Post-Reaction
Characterization of Technical
Catalyst Formulations

Bio-Oil Thermal
Stability
Assessments

Technoeconomic and Lifecycle Analysis



Approach: Management Plan

Task 1: Project Management and Industry Engagement

Lead: Mike Griffin

Task 2: Catalytic Fast Pyrolysis of Biomass

Lead: Cody Wrasman

Task 3: Hydroprocessing of CFP-Oil to SAF

Lead: Kristiina Iisa

Task 4: Thermal Stability of CFP-Oil

Lead: Mark Nimlos

Task 5: Characterization of Catalyst Properties

Lead: Susan Habas

Task 6: Refinery Economic and Optimization Modelling

Lead: Michael Talmadge

Diversity, Equity, and Inclusion Lead: Anne Starace

- Dedicated funding to support DEI efforts
- DEI-position on the project management team
- Inclusion of DEI milestones as project deliverables



Partnering with Minority Serving Institutions



Supporting
Minority Owned
Businesses



Engaging through workshops and 'DFI-minutes'



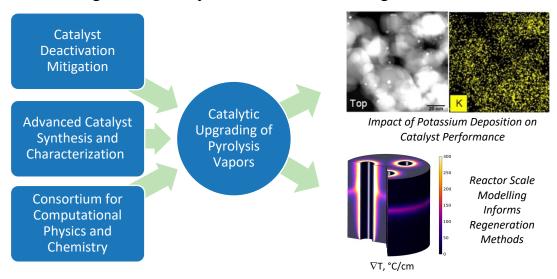
Collaborating on 'JUST-R' Metric Development*

*Dutta, N. S., et al., "Just-R metrics for considering energy justice in early-stage energy research", Joule, 2023, in press



Approach: Communication and Collaboration

Coordination with Enabling Projects Provides Fundamental Insight into Catalyst Deactivation and Regeneration



<u>6 Collaborative Manuscripts with Enabling Projects Since 2021</u>

Adkins, B., et al., *Reaction Chemistry & Engineering*, **2021**Unocic, K.; et al. *Microscopy and Microanalysis*, **2021**, 27, 796-798
Unocic, K., et al. *Journal of Visualized Experiments*, **2021**, 173
Lin, F., et al., *ACS Catalysis*, **2022**, 12, 1, 465-480
Downes, C. A., et al., *Chemistry of Materials*, **2022**, 34, 6255
Unocic, K. A., et al., *Microscopy and Microanalysis*, **2022**, 28, 2472

Industry Engagement Promotes Commercially Impactful R&D Outcomes

Johnson-Matthey

CRADA: Accelerating CFP catalyst and process development through collaborative R&D

ExxonMobil

CRADA: Advancing biomass pyrolysis technologies through collaborative R&D

Phillips 66

Project-level collaboration focused on hydroprocessing of biogenic oils

ChemCatBio Industrial Advisory Board

Consortium level guidance related to the conversion of biomass and waste carbon

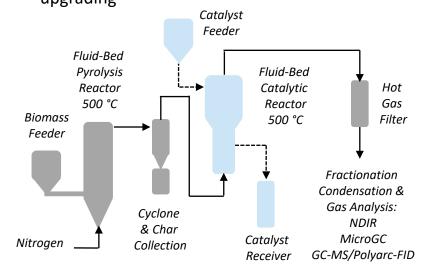


Progress and Outcomes: Catalytic Pyrolysis

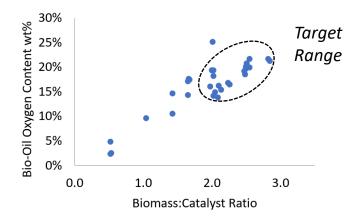
Progress

 Commissioned a fluidized bed reactor system with continuous pyrolysis and catalytic upgrading capabilities

 Completed an experimental campaign to determine product yield and composition at varying levels of catalytic upgrading



Feedstock: 50/50 Clean Pine + Forest Residues Catalyst: ZSM-5 SAR 30 with Alumina Binder



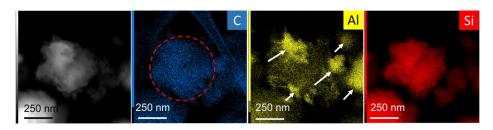
CFP-Oil Properties				
B:C, g/g	1.7	2.5	2.8	
Carbon Efficiency, %	26	30	31	
Oxygen Content, wt% db	17	20	22	
Carboxylic Acid Number, mg KOH/g	19	29	30	
Sulfur, ppm	32	32	27	

- Established benchmark product yield and composition data for CFP
- Achieved carbon balance closures of 100±5% through comprehensive analysis of the feedstock and product streams
- Generated three 500+ mL samples of CFP-oil with oxygen content ranging from 17-22 wt%

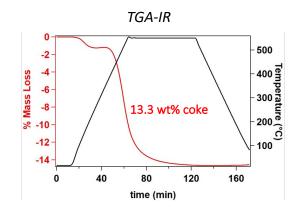


Progress and Outcomes: Catalyst Characterization

Progress: Applied physical and chemical characterization techniques to evaluate deactivation mechanisms and inform regeneration procedures for CFP catalysts



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Sample	Surface Area (m²/g)	Acid Site Density (μmolNH ₃ /g)
Fresh Catalyst	340	765
Post Reaction	51	516
Regenerated	336	722

Outcomes:

- Generated benchmark characterization data for technical zeolite catalysts with an emphasis on determining the unique composition and combustion properties of biogenic coke
- Confirmed efficacy of oxidative regeneration procedure to restore CFP catalyst active sites
- Ongoing: Pre- and post-reaction characterization of hydrotreating catalysts

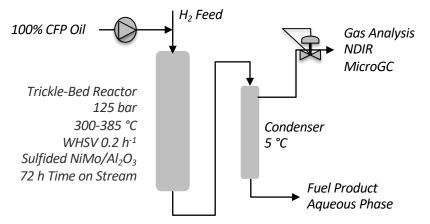
Ongoing: Experimental/computational collaboration with CCB enabling projects to evaluate the composition, distribution, and combustion kinetics of biomass-derived coke (CFP, CDM, ACSC, CCPC)



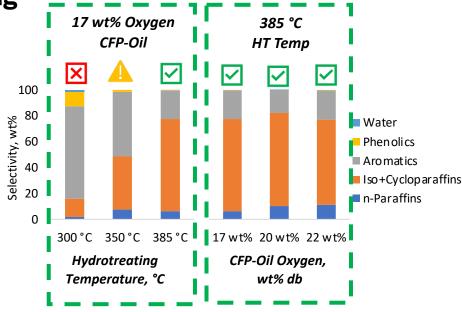
Progress and Outcomes: Hydrotreating

Progress

Completed an experimental campaign to determine the impact of CFP-oil oxygen content and hydrotreating temperature on product yield and composition



Hydrotreating Results, 385 °C				
CFP-Oil Oxygen Content	17 wt%	20 wt%	22 wt%	
HT Carbon Efficiency	91%	92%	89%	
Hydrogen Consumption	7%	8%	13%	
HT-Oil Oxygen Content	<0.001 wt%	<0.001 wt%	<0.001 wt%	
HT-Oil H:C Ratio	1.76	1.76	1.81	



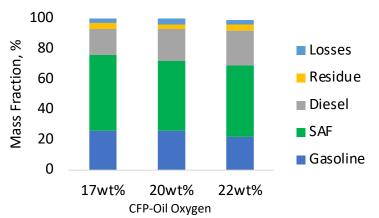
- Established benchmark yield and composition data for stand-alone hydrotreating
- Identified conditions suitable for the production of a high-quality hydrotreated oil with oxygen levels below detection limits

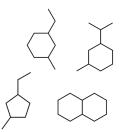


Progress and Outcomes: Fractionation and Fuel Properties

Progress

Fractionate the hydrotreated oils into gasoline, SAF, and diesel cuts using a micro-spinning band distillation system





>75 wt% Cycloalkanes in SAF Fraction

- Primary component in Jet A
- Difficult to access via other SAF pathways (HEFA, FT, ATJ)
 - Increased energy density and cleaner burning than aromatics

SAF Properties					
CFP-Oil Oxygen Content, wt% dry basis	17 wt%	20 wt%	22 wt%		
Density @15°C, 0.730-0.880 g/ml	0.854	0.843	0.847		
Flash Point >38 °C	41.5	41.5	39.5		
Freezing Point, <-40 °C	<-80	<-80	<-80		
Surface Tension 22°C, 25-29 mN/m ^b	28	27	28		
Lower Heating Value, >42.8 MJ/kg	42.5	42.7	42.6		
D86 Simdis T10 150-205 °C	162	162	161		
D86 Simdis FBP <300 °C	253	250	254		

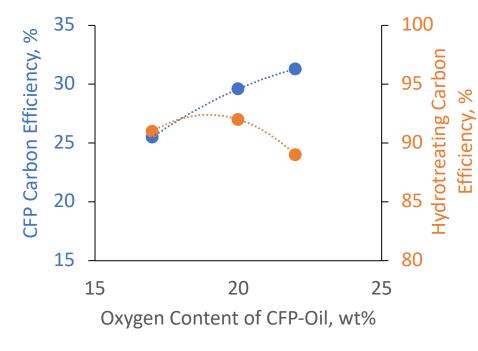
- Established benchmark yield and compositional data for gasoline, diesel, and SAF fractions
- Generated a cycloalkane-rich SAF product that meets key ASTM D4054 guidelines for fuel properties

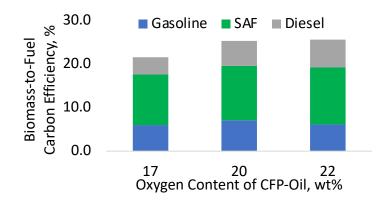


Progress and Outcomes: Yield Structures

Progress

Determined carbon efficiencies and compositional data for the integrated biomass-to-fuels process





Preliminary TEA/LCA: Thermochemical Process Analysis 2.1.0.302

Oxygen in CFP Oil, wt%	17%	20%	22%
Estimated Minimum Fuel Selling Price	6.1-7.5	5.7-6.9	5.3-6.5
Estimated GHG Reduction, %	84	78	75

¹ Modelled *Minimum Fuel Selling Price* (MFSP) in 2016\$, with ±10% variation

- Revealed correlations between process yield and bio-oil oxygen content for CFP and hydrotreating steps
- Provided experimental data to support technoeconomic and lifecycle analysis

²Approximate GHG reduction over petroleum gasoline (93 g CO₂e/MJ) with coproduct electricity from usable excess energy not converted to liquid fuels.

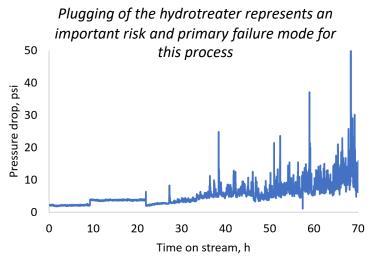


Progress and Outcomes: Critical Material Attributes

Progress

Identified CFP-oil compound classes with potential links to system plugging during hydrotreating

Compound Class	Proposed Plugging Mechanisms	Critical Material Attribute (CMA)	Detection Methods
Reactive Carbonyls	Condensation Reactions	HO O	GCMS
Anhydrosugars	Cross-Linking & Gelation	OH OH Levoglucosan	GCMS
High Molecular Weight Oligomers	Viscosity & Nucleation	> 500 Dalton Heavies	GPC



Data were collected during experiments performed under a collaborating project: 3.2.1.005 "Advancing the Development of Biofuels for the Maritime Sector"

Outcome

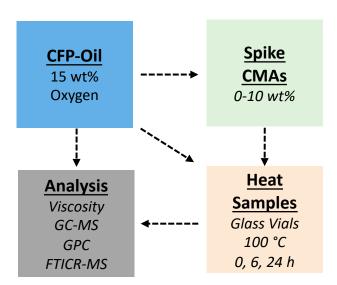
Down-selected three critical material attributes (CMAs) for further analysis via chemical spiking and thermal stability experiments



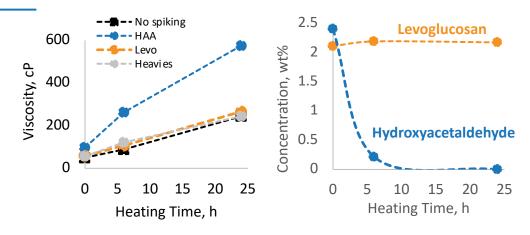
Progress and Outcomes: Critical Material Attributes

Progress

Completed a series of chemical spiking experiments to evaluate the impact of CMA concentration on thermally-driven increases in viscosity and molecular weight



High Throughput; < 10 mL Material Requirements



Outcomes

- Identified correlations between hydroxyacetaldehyde (HAA) concentration, viscosity, and molecular weight growth
- Identified reactive carbonyls as a primary species of concern

Ongoing: Follow-on thermal stability experiments and statistical analysis to further probe correlations between concentration, viscosity, and molecular weight for reactive carbonyls



Atmospheric and Vacuum Distillation

Impact: Pathway to Market

Refinery Hydroprocessing Delayed Coking Fluid Catalytic Cracking Hydrotreating Hydrotreating

Alkylation

Kerosene/Jet Fuel

Gasoline Hydrotreating Isomerization

Reforming

Gasoline

Refinery integration points with direct pathway to SAF

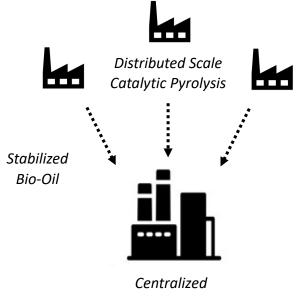
Diesel

Hydrocracking Dieses Hydrotreating

Product Blending and Sales

- 100+ BGPY current US hydrotreating/hydrocracking capacity
- Leverages existing workforces and institutional knowledge
- Potential for blending multiple biogenic and fossil streams

Hub and Spoke Production Model



Centralized
Refinery Hydroprocessing

US 2022 Refinery Capacity Summary			
Diesel Hydrotreater 70 BGPY			
Hydrocracker 37 BGPY			

https://www.eia.gov/petroleum/refinerycapacity/



Impact: Scientific Knowledge



12 Peer Reviewed Publications Since 2021

See Supporting Slides



12 External Presentations Since 2021

See Supporting Slides



2 Issued Patents 6 Pending Applications

Novel catalysts, processes, and co-products



Synthesis and Characterization of Technical Catalysts

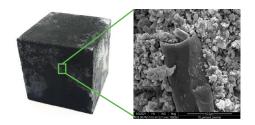
CCB Focus Area

Spin-Off Projects Since 2021

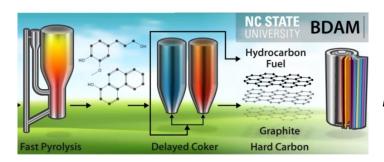
High-Performing Carbon-Negative Concrete Using Low-Value Byproducts From Biofuels Production



Carbon Upcycling
Colorado School of Mines
University of Colorado Boulder



Biocrude Derived Anode Materials for Lithium-Ion Batteries



NC State University
Birla Carbon
Yale University
Battery Innovation Center
Ensyn Corporation



Approach

- **Supports BETO SAF production goals** through integrated pathway development that reduces technical risk, addresses feedstock constraints, and informs process scale-up
- Active project management with integrated risk identification and mitigation efforts as well as a dedicated DEI effort
- Established avenues for coordination with multi-lab collaborators and industrial partners

Progress and Outcomes

- Established benchmark yield structures and compositional data for three levels of CFP upgrading
- Provided first-of-its kind **evaluation of SAF fuel properties** and identified opportunities to produce a cycloalkane-rich product stream
- Generated benchmark **characterization data for technical zeolite catalysts** with an emphasis on determining the unique composition and combustion properties of biogenic coke
- Established bio-oil critical material attributes to mitigate the risk of plugging during down-stream hydroprocessing

Impact

- Informed the development of **TEA/LCA process models** through the generation of high-quality data sets
- Generated **broadly enabling scientific knowledge and multiple spin-off projects** to advance the state of technology
- Aligned with a promising pathway to market that addresses an emerging demand for biogenic oils



Acknowledgements







Cody Wrasman



Mark Nimlos



Susan Habas



Michael Talmadge



Anne Starace

Trevor Smith (BETO) Steven Rowland (NREL) Calvin Mukarakate (NREL) Nolan Wilson (NREL) Abhijit Dutta (NREL) Joshua Schaidle (NREL) Fred Baddour (NREL) Brittney Petel (NREL) Nicole LiBretto (NREL) Matt Yung (NREL) Kellene Orton (NREL) Tyler Cary (NREL) Rianna Martinez (NREL)

Scott Palmer (NREL) Carson Pierce (NREL) Renee Happs (NREL) Earl Christensen (NREL) Robert McCormick (NREL) Alexander Rein (NREL) Andy Young (NREL) Xiaolin Chen (NREL) Cheyenne Paeper (NREL) **Huamin Wang (PNNL)** Fan Lin (PNNL) Kinga Unocic (ORNL) Biva Talukdar (ORNL)







This work was performed in collaboration with the Chemical Catalysis for Bioenergy Consortium (ChemCatBio, CCB), a member of the Energy Materials Network (EMN)

U.S. DEPARTMENT OF Office of ENERGY EFFICIENCY & RENEWABLE ENERGY BIOENERGY TECHNOLOGIES OFFICE

Questions

chemcatbio.org

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Additional Slides



Timeline 10/1/21- 9/30/24

	FY22 Costed	Total Awar
DOE Funding	1.7 MM + 300 k Carryover	2.0 MM

TRL at Project Start: 3 TRL at Project End: 4

Project Goal

The objective of this project is to produce sustainable aviation fuel (SAF) from lignocellulosic biomass through catalytic fast pyrolysis (CFP) and hydrotreating

End of Project Milestone

Perform a series of integrated CFP and hydrotreating experiments to achieve a \geq 20% relative increase in carbon efficiency to jet-range products compared to the baseline established in FY22. The CFP-oil will meet the CMAs and tolerance ranges identified previously, and the SAF fraction will meet ASTM4054 specifications for density, viscosity, boiling point, heating value, flash point, freeze point, and volatility. Hydroprocessing durability will be demonstrated via experiments performed in a realistic environment for a minimum of 500 h time on stream. Technoeconomic and lifecycle analysis will be performed to determine the minimum jet selling price and demonstrate a \geq 70% reduction in GHG emissions compared to fossil-based pathways.

Funding Mechanism

FY22 Lab Call

Project Partners

- ExxonMobil (CRADA)
- Johnson Matthey (CRADA)
- Phillips 66 (Hydrotreating Advisors)

TEA/LCA— Additional **Preliminary** Information

Financial assumptions consistent with FY20-21 State of Technology report*

Description of Assumption	Assumed Value
Cost year	2016
Internal rate of return on equity	10%
Plant financing by equity/debt	40%/60% of total capital investment
Plant life	30 years
Income tax rate	21%
Interest rate for debt financing	8.0% annually
Term for debt financing	10 years
Working capital cost	5.0% of fixed capital investment (FCI) (excluding land purchase cost)
Depreciation schedule	7-year MACRS a schedule [9]
Steam plant depreciation	20-year MACRS schedule [9]
Construction period (spending schedule)	3 years (8% Y1, 60% Y2, 32% Y3)
Plant salvage value	No value
Startup time	6 months
Revenue and costs during startup	Revenue = 50% of normal Variable costs = 75% of normal Fixed costs = 100% of normal
Onstream percentage after startup	90% (7,884 operating hours per year)

^a Modified accelerated cost recovery system

CFP Oil Oxygen Contents →	17%	20%	22%
Feedstock cost* (\$/dry US ton)	67	67	67
Capital investment total (million \$)1	707	728	740
Chemical coproducts	No	No	No
Electricity credit (cents/GGE)	78	57	45
MFSP ^{2,3} (\$/GGE)	6.1-7.5	5.7-6.9	5.3-6.5
GHG Reduction over gasoline (%)	84	78	75

¹ Capital and operating costs based on the ex situ case in the 2015 design report (https://www.nrel.gov/docs/fy15osti/62455.pdf). Current model at lower pressure than 2015 report, leading to larger equipment sizes and higher capital costs for CFP equipment. ² Model yields considered hydrotreated products heavier than diesel and other losses during product distillation as fuel products because the heavy ends from distillation are hydrogenated and stabilized, and can likely be used as marine fuel

^{*}Reference: https://www.nrel.gov/docs/fy21osti/80291.pdf; GGE: gallon gasoline equivalent

³ CFP catalyst replenishment is based on values in the 2015 design report (based on typical FCC values). The reactor heat balance is used to determine the flow rate. With a less active catalyst (reflected by different biomass to catalyst ratios in our experiments), cheaper catalyst diluents can allow CFP catalyst cost reduction. NREL | 22

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French, R. J.; lisa, K.; Orton, K. A.; Griffin, M. B.; Christensen, E.; Black, S.; Brown, K.; Palmer, S. E.; Schaidle, J. A.; Mukarakate, C.; Foust, T. D., Optimizing Process Conditions during Catalytic Fast Pyrolysis of Pine with Pt/TiO₂—Improving the Viability of a Multiple-Fixed-Bed Configuration. *ACS Sustainable Chemistry & Engineering* **2021**, 9, 3, 1235-1245.

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